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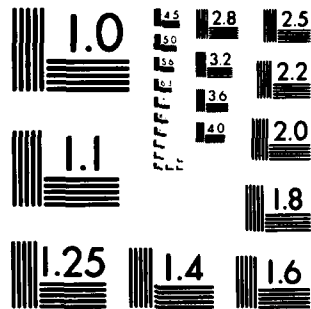
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USE OF GALLIUM TRIPLE POINT AS HIGH TEMPERATURE REFERENCE

A. EDWARD GENT and
ANDREW GUESS

January 1982

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FOREWORD

The effort described in this report was performed by personnel in the Maintenance Engineering Division of the Engineering Department, Naval Oceanographic Office (NAVOCEANO). This work was performed to evaluate and document a new and unique capability within NAVOCEANO for providing precision temperature calibrations of oceanographic survey sensors. If further information or discussion on this topic is desired by the reader, contact Commanding Officer, Naval Oceanographic Office, attention Mr. Adolph H. Klein, Maintenance Engineering Division, NSTL Station, Bay St. Louis, MS 39522, 601-688-4465 (FTS 494-4465, AV 485-4465).



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Captain, U. S. Navy
Commanding Officer

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>> Evaluation of a triple point of gallium cell for use as a primary standard in checking the accuracy of laboratory platinum resistance thermometers is described. This device allows PRT's to be checked at the upper range of interest in oceanographic temperatures. The temperature specification for the gallium triple point cell is 29.7739 \pm .0001$^{\circ}$C.</p>		

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INTRODUCTION

In an effort to enhance quality control in precision temperature calibrations of oceanographic and hydrographic survey instruments, a Triple Point of Gallium standard was built for the Naval Oceanographic Office (NAVOCEANO) under contract by the National Bureau of Standards. The new primary standard provides a temperature reference point (29.7739°C) for calibration verification of platinum resistance thermometers (PRT), which are used as laboratory secondary standards. PRT calibration checks at the triple point of gallium allow accuracy verification at the high temperature end of the typical oceanographic operating range (from -2 to 30°C). Present PRT calibrations are performed at the triple point of water ($.01^{\circ}\text{C}$), which allows for correction of offset. The gallium cell permits a two point calibration of PRTs providing a means for determining shifts in calibration curve slope as well as offset over the range of interest.

In July 1979, the National Bureau of Standards (NBS) delivered the gallium triple point cell for use at NAVOCEANO's Sensor Calibration Laboratory. This cell, shown in figure 1, is a sealed cylindrical cannister constructed of steel with an inner lining of nylon and teflon. The cell contains 900 grams of 99.9999% pure gallium held under vacuum. A thermometer well for PRT insertion protrudes into the cell along the cell's axial center.

NBS has determined that the gallium triple point cell constructed for NAVOCEANO is $.0001^{\circ}\text{C}$ lower than the triple point of pure gallium and has certified the cell's triple point to be $29.7739 \pm .0001^{\circ}\text{C}$.

CELL PREPARATION

The triple point of gallium is obtained using a procedure outlined by NBS in which an immersion bath is temperature controlled above the triple point temperature of gallium. The cell is prepared by first soaking in an ice bath for 12 to 14 hours. This completely solidifies the gallium mass. The cell is then submerged in a hot bath at 50 to 60°C . A copper rod is used to force the hot water into and out of the thermometer well. This procedure is continued up to a maximum of six minutes. This hot soak technique is used to melt a small amount of the gallium mass around the periphery of the container walls and also around the PRT well. Holding the cell upright it is given a sudden twist, thus breaking the solid mantle free within the cell. An alcohol-deionized water solution, used as a heat transfer medium, is poured into the thermometer well, filling about one inch of the bottom of the well. At this point, the cell is placed in a specially constructed temperature controlled bath with a setpoint stability of $\pm .008^{\circ}\text{C}$. This bath, shown in figure 2, consists of a rectangular insulated tank with a motor driven stirrer, a Tronac PTC 40 controller, and hinged lid. The gallium cell is positioned within the bath in a manner such that the thermometer well is aligned with an access hole in the lid for insertion of a PRT. The bath temperature is adjusted slightly above the triple point temperature of the gallium allowing a slow change of phase of the mantle from solid to liquid to occur.

TEST PROCEDURE

In order to evaluate the stability of the gallium cell at its triple point of 29.7739°C, the following equipment was used. Two Leeds and Northrop Platinum Resistance Thermometers, Type 8167-25 with serial numbers 1797882 and 1797885 and two Guildline Voltage Ratio Bridges, Model 9975, bearing serial numbers 44032 and 48330.¹ The PRTs were mated with a particular bridge throughout the test with Unit 1 consisting of PRT #1797885 and bridge #44032 and Unit 2, PRT #1797882 and bridge #48330. All measurements were made with a .001 ampere bridge current. Temperature measurements on the cell were taken daily and new R_0 values were obtained throughout the test. R_0 refers to the measured resistance value of the PRT at the triple point of water.

TEST RESULTS

Gallium cell measurements were conducted during the period May 11 to October 1, 1981. After several attempts were made to find a bath temperature suitable for maintaining the gallium at its triple point for long periods, a bath temperature of 29.7840°C \pm .008°C produced a stable triple point lasting 64 days. During this period, daily PRT measurements indicated gallium cell stability to be \pm .0001°C for a given PRT R_0 . PRT measurements for Unit 1 ranged from 29.77338°C to 29.77418°C and Unit 2 from 29.77223°C to 29.77184°C. Temperature variations resulted from R_0 changes in each PRT. However, these temperatures are within the \pm .002°C uncertainty of the PRT measurement about the gallium triple point of 29.7739°C. Cell stability began to degrade after the 64th day. The gallium cell was recycled and a second test was performed with similar results. The cell remained stable for 65 days, after which the testing was concluded.

SUMMARY

The tests conducted have shown the feasibility and reliability of using the triple point of gallium as a reference point in maintaining the accuracy of PRT standards used in calibrating oceanographic sensors. Efforts are currently underway to incorporate the gallium triple point in the PRT calibration process to assure the accuracy of measurements at high temperature (30°C). With the success of the gallium triple point cell, an investigation into a triple point system, near 15°C will be pursued to provide a mid-range linearity check point. Such a device will allow an unprecedented three point check of PRT standards over the range of interest to oceanographic investigators.

1. The temperature uncertainty using this measurement is \pm .002°C.

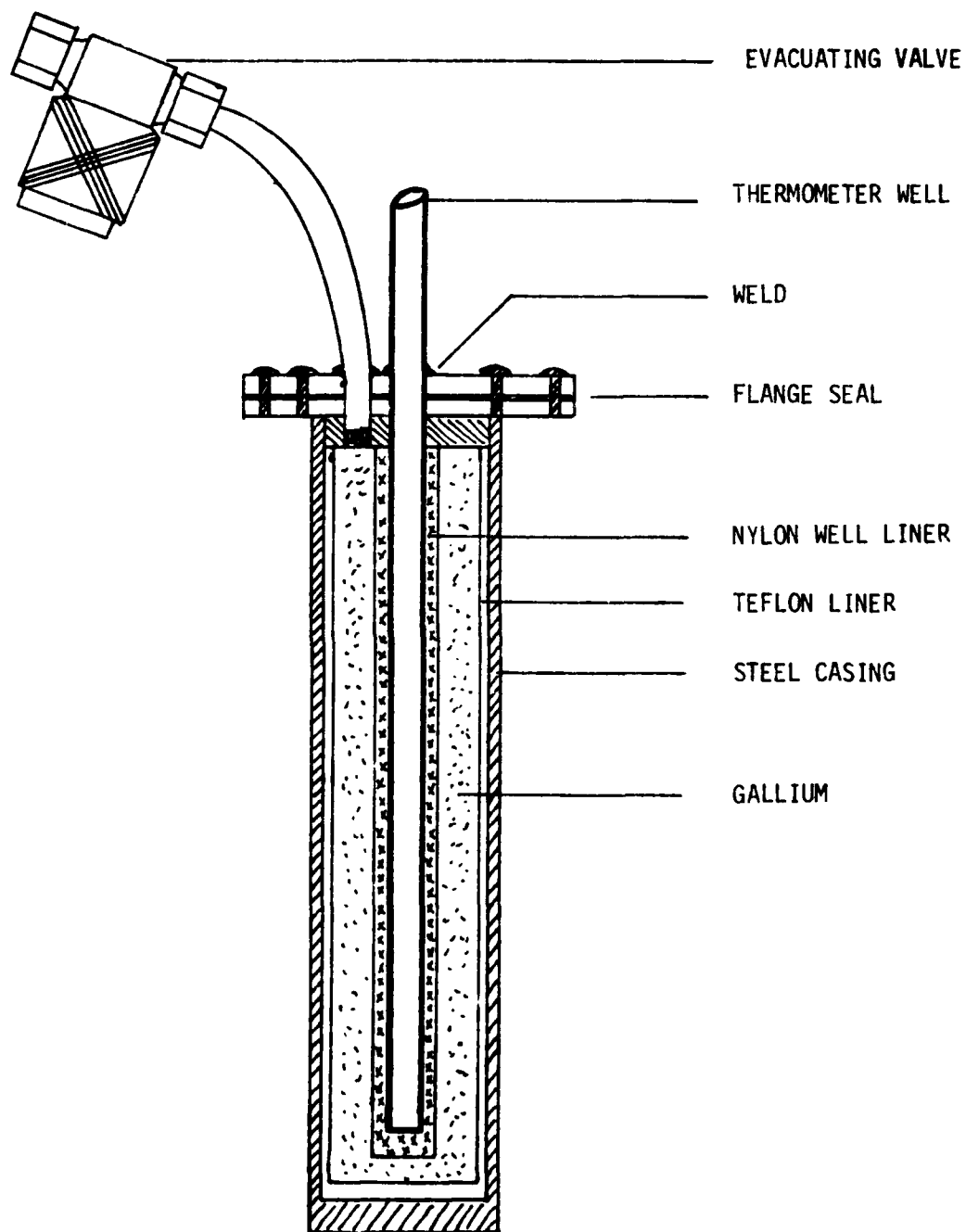


FIGURE 1 - GALLIUM TRIPLE-POINT CANISTER

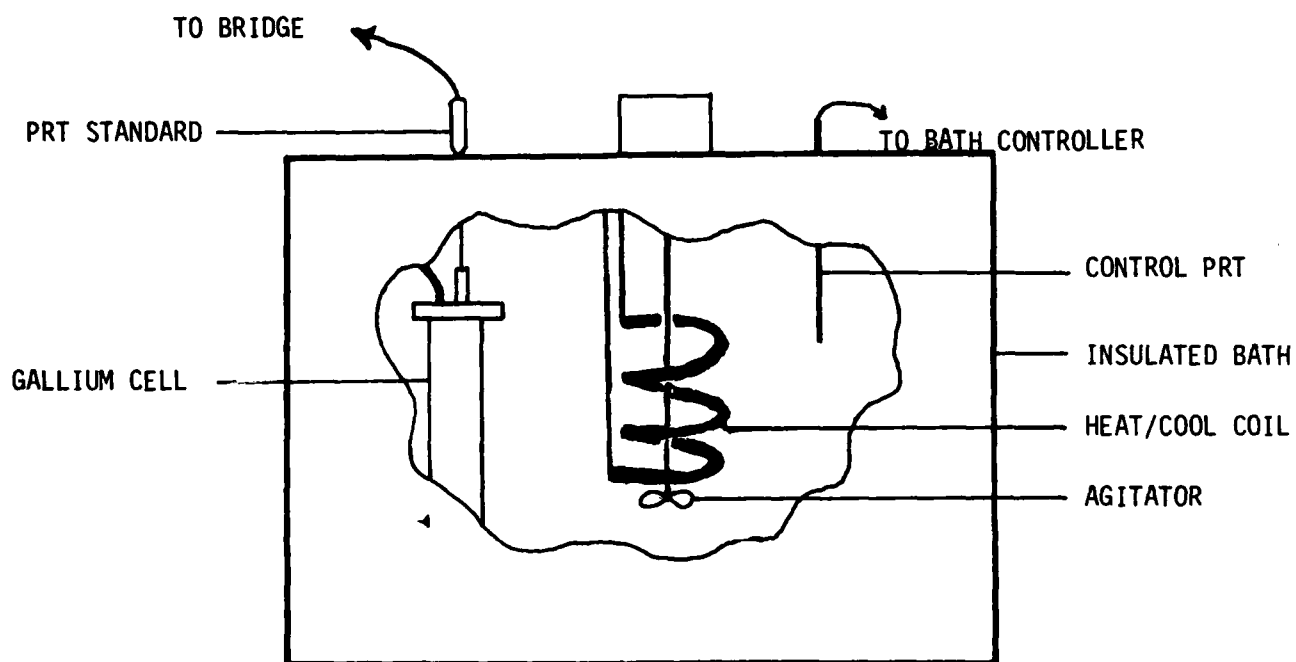


FIGURE 2 - GALLIUM TRIPLE POINT BATH

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